STATE OF THE ART

AND

FUTURE DIRECTIONS IN SOLVENT EXTRACTION

PROCEEDINGS
OF THE

FIRST INTERNATIONAL
SOLVENT EXTRACTION WORKSHOP'97
OCT. 5-8, 1997
BANFF, ALBERTA, CANADA

Organized and Chaired by Dr. Gordon M. Ritcey

Sponsored by the International Committee for Solvent Extraction Technology (ICST)

CONTENTS

FORWARD

The following is a summary of the Solvent Extraction Workshop'97 which was held October 5-8, 1997 in Banff, Alberta, Canada.The Workshop, probably the first of its kind covering all aspects of the Solvent Extraction process, was by invitation only.

For some background to the Workshop, at the ISEC'96 in Melbourne, I proposed the plan to hold a "small meeting of Solvent Extraction specialists" sometime between the Melbourne meeting and the next ISEC'99 in Barcelona in 1999. The purpose would be to bring together many of the experts in solvent extraction to identify and to discuss the various problem areas in this unit process. The recommendations would provide the many researchers throughout the world with good project ideas that could result in significant reductions in capital and operating costs of solvent extraction plants. There was agreement from the International Solvent Extraction Committee (ICSE), and others, on such a workshop. After considering several venues, I decided to hold this initial meeting in Banff, Alberta, Canada.

This Workshop was planned not as the usual, formal, conference, but instead was styled after the very successful and informal Gordon Research Conferences that have been held in the USA on various aspects of chemistry and engineering. Usually, those meetings are by invitation only, and this was the case with the Solvent Extraction Workshop.

The list of possible invited experts for such a Workshop was assembled by members of ICSE, and if some have been overlooked, it was not intentional. If the Workshop was to be successful, the total attendees would be limited to about 75.

So it was hoped that the invited specialists would recognize that their participation would be useful to the solvent extraction community in general, as well as an opportunity to personally benefit from the meetings. Invitees included those from all aspects of chemistry, chemical engineering, process design, engineering contractors, and operating plants. It was anticipated that the Solvent Extraction Workshop of "experts" in the areas of R & D, engineering and applications. would:

1) critically review the individual areas of concern in the consideration of solvent extraction -- from R&D to its industrial scale-up to plant processing;

2) provide a closer interaction between chemistry-engineering-process development-plant design and construction then is possible in the usual, large international meetings;

3) improve the communication between the R&D scientists and engineers and the engineering contractors to optimize the design of future solvent extraction operations;

4) transfer the conclusions of this meeting to ISEC'99, thus providing some guidance to future R&D and plant design; and,

5) to provide a document for wide circulation amongst practitioners, possibly published in Hydrometallurgy or Solvent Extraction and Ion Exchange.

The meetings took place over a 4-day period, although the first day, Sunday, was really to arrive and meet each other socially at a Reception, followed by dinner.

The format comprised an informal technical session in the morning, followed by lunch and then break for the afternoon. This permitted the attendees to visit the area for sightseeing, or participate in sporting activities of their choice. Following dinner, the evening sessions commenced.

The first day, Monday, the morning and evening sessions had all the participants together for invited keynote presentations. Following those presentations, during the first-half of the next morning, Tuesday, an open forum of the group established an extensive list of "concerns" that could be identified as critical in the solvent extraction process--from the chemistry and chemical engineering, through to the process and plant designs, engineering and plant operations. The subsequent Group Workshops that morning and Tuesday evening and Wednesday morning dealt further with the list of concerns in more detail, thus providing ideas for R&D and engineering required in that area. The Workshop closed Wednesday afternoon with the summary presentations by the groups to the total Workshop meeting.

Like any technical meeting, there were a number of invitees who were unable to attend due to other commitments, which was indeed unfortunate. However, those that did attend were rewarded, I believe, in the useful discussions that resulted with their participation. The venue of the Banff Centre was perfect, and the weather cooperated--with 2 glorious days of sun and warmth in the mountains, followed by 2-days of snow which transferred the autumn scenery to a white fairyland of snow-covered landscape. And we also had the presence of numerous deer which chased a few of us!

Gordon M. Ritcey, SX Workshop'97 Chairman

AGENDA

Sun. Oct.5

1515-1715	REGISTRATION
1730-1830	RECEPTION
1830	BUFFET DINNER

Mon. Oct.6

0700-0900	BUFFET BREAKFAST
0730-0815	REGISTRATION

<u>SESSION #1 PLENARY SESSION - KEYNOTE PRESENTATIONS</u>

0830 OPENING REMARKS .... G.M. RITCEY

0845 NEW REAGENTS OR NEW WAYS WITH OLD REAGENTS .... M. COX/D.FLETT

0900 REAGENT SYNTHESIS .... M.VIRNIG

0915 SYNERGISM .... H. ALY

0930 REACTIVITY/REAGENT BREAKDOWN .... J. SZYMANOWSKI

0945 EXTRACTION CHROMATOGRAPHY
(SOLVENT IMPREGNATED RESINS) .... P. HORWITZ

1000 INTERFACIAL REACTIONS .... H. WATARAI

1015 BREAK

1045 MICROEMULSIONS/MICELLES .... S. ORTIZ

1100 CHEMICALLY ACTIVE ADSORBENTS .... L. TAVLARIDES

1115 MASS TRANSFER & EQUILIBRIUM DATA .... H. BART

1200 BUFFET LUNCH

AFTERNOON FREE

1700-1900 DINNER

AGENDA (continued)

SESSION #2 PLENARY SESSION - KEYNOTE PRESENTATIONS

1900 EQUIPMENT AND TRENDS .... M. SLATER

1915 MIXER SETTLERS .... M. VANCAS

1930 CFD FOR SX EQUIPMENT DESIGN .... H. BART

1945 INTERFACIAL PHENOMENA .... G. STEVENS

2000 DISPERSION AND COALESCENCE .... S. HARTLAND

2015 HYDRODYNAMICS IN MIXER SETTLER .... G. MILLER

2030 BREAK

2100 CENTRIFUGAL EXTRACTORS DEVELOPMENT
AND UTILIZATION IN INDUSTRY .... A. PUSHKOV/G. KUZNETSOV

2115 STUDY AND APPLICATION OF EXTRACTORS IN CHINA .... W.Y. FEI

2130 APPLICATION OF BATEMAN PULSE COLUMN FOR
URANIUM EXTRACTION .... R. KLEINBERGER

2145 WASTE MANAGEMENT-RADIOACTIVE WASTES .... T.TODD

2200 MECER - AN ON-LINE METAL RECOVERY PROCESS -
TECHNICAL AND ECONOMIC ADVANTAGES .... H.REINHARDT

2215 COMPARISON OF SULPHATE AND
CHLORIDE EXTRACTION .... V. LAKSHMANAN

2230 PHYSICAL-CHEMICAL ASPECTS (EMULSIONS/CRUDS) .... G. RITCEY

WORKSHOP SESSIONS
TUE MORNING AND EVENING SESSIONS;
AND WED MORNING & AFTERNOON SESSIONS

WORKSHOP SUMMARY PRESENTATIONS

WED OCT 8 1330

This Final Session will be held immediately following lunch, instead of in the evening. This will give those that depart on Wednesday evening the opportunity to participate. This Session will be devoted to summary presentations of the individual work groups to the attendees.

1630 CLOSE OF WORKSHOP

OPENING OF CONFERENCE - Dr. Gordon M. Ritcey

Good morning friends, and welcome to Banff and to the SOLVENT EXTRACTION WORKSHOP'97. I am pleased that so many are here who I have known for quite some time, and whose expertise I have great respect. Although there have been many short courses, conferences and seminars on solvent extraction in the past, as well as seminars directed to such as copper, cobalt, uranium, I believe this is the first Workshop on the complete aspects of SX-- from the basic studies in chemistry and chemical engineering, through process design, piloting, plant design, scale-up and operation.

This workshop has been a long time in the "thinking" process. In many discussions with the late Carl Hanson as I hiked with him in the Yorkshire Dales we concluded in 1980 that it was time to have such a meeting of experts, for we believed that optimization of design would eventually catch up with us, and we might not have the sufficient answers to provide the engineers that would design and construct the SX plants. We thought that 50 should be the ideal number of participants, and the meeting would be held in the UK. We believed that a venue away from cities would be preferred, and so we decided that a hotel in Witby, near the sea in Yorkshire, would be an ideal location. However, Carl subsequently was unwell, and passed away before the remainder of the planning could be completed.

The years passed, and I finally pursued the idea and presented it to the International Committee for Solvent Extraction Technology during the ISEC'96 meeting in Melbourne. Withe the support of that Committee, planning for such a meeting resumed. The real work commenced on the Workshop in January 1997.

The choice of a venue for this meeting in Canada came down to either the small town of Digby on the Bay of Fundy on the east coast, or in the mountains of Banff in the west. Economics dictated Banff, and I am sure it will have been a good choice. So I hope that for those who are in Banff for the first time, as well as others who have had the opportunity to visit before, that you will enjoy the beautiful scenery that the area offers.

The decision for this meeting was to limit the number of participants in the range of 75. If there had not been some last minute cancellations by a few who were unable to attend for several reasons, our registration would have been just over 80, instead of about 70. Nevertheless, a wide spectrum of expertise and background are here, representing 21 countries. I therefore hope that everyone participates in the discussions, and thus benefit from the valuable interaction in the Workshops.

Solvent Extraction is a unit operation practised for the recovery of many substances. As such, we would refer to it as an applied science and not of only fundamentals of chemistry and chemical engineering, nor only the practical areas of process design and engineering through to operation. To be successful in the overall development of the final operating plant, a number of disciplines as shown below, will contribute to a cost-effective operation.

The OBJECTIVES of the Workshop are:

1) To address and critique the various areas of the solvent extraction process, including:

- A) Fundamental research in chemistry and chemical engineering
- B) Flowsheet development (chemistry, chemical engineering, metallurgy) through to piloting to obtain preliminary design and cost data
- C) Engineering design, control, plant design and plant construction
- D) Plant operation (problems and solutions)

2) To determine where there are gaps or flaws in our knowledge so that:

- A) Additional fundamental research projects would be identified and carried out;
- B) Possibly some on-going fundamental research may be identified as not generally applicable to improving the process in the long term;
- C) Identify where improved chemistry/extractants/contactors and control could be developed or improved to enhance the overall operation and economics in the process and plant design; and,
- D) An improved understanding and communication between chemists-engineers-engineering companies will result in improved plant operations, thus reducing the many costly operating problems of plants.

So how are we to achieve the objectives?

The Workshop is informal and hopefully more like a "think tank; but still with some structure.

Each of the Workshop sessions is comprised of a number of items to address-- but not necessarily limited to those subjects. Each Session will have chairpersons. The Sessions will therefore be guided from one subject to the next by the chair-persons. As in any "think tank" type of discussion, the questions and answers gradually will arrive at a point where "feeding on one another" will have provided some real information and perhaps, even conclusions.

Of course in a single 3-day meeting we can't expect to solve all the problems, or determine all the gaps requiring further research. But we can make a start in that direction, and at the same time gain valuable information in the subject of interest.

However unless we make some sort of record of our discussions--we'll have done only half of the job. To achieve the objectives of defining R&D needs we must have some conclusions. So that is why there are chairpersons to ensure that the final conclusions on each subject area of their session are noted. At the same time, hopefully each session will be taped (no names of individuals comments!) so that possibly a more complete document of our Workshop can be described. Such a report could be published, as well as presented at ISEC'99 in Barcelona. Research could then, through funding, begin to close the gaps in our understanding.

Finally, in the wrap-up Plenary Session on Wednesday afternoon, representatives from each of the Workshop Sessions would present a summary to the total participants of the Workshop. Since each of the discussion areas are connected, as shown in the diagram of the SX process, I would expect that such summaries will provide information to all the participants.

INVITED PRESENTATIONS

A number of invited keynote presentations took place on the first day of the Workshop. The speakers and their subjects are given in the Agenda. The objects of these short presentations of about 15 minutes was to provide an "overview" or state-of-the-art" of a particular area in the solvent extraction process. These presentations therefore were helpful in setting the stage for the subsequent in-depth discussions that took place the following two days. The texts of those presentations are not included here, but can possibly be obtained by contacting the authors directly.

GENERAL WORKSHOP CONCERNS/RECOMMENDATIONS

An open discussion of all the participants identified the following areas of concern which would subsequently, where time permitted, be discussed in more detail in the smaller workshop groups. It was left to each separate working group to select from the list of concerns below those that they could discuss in the time available for the Workshop. It should be noted that some items were addressed by more than one group, and often with similar statements of concern. There was no attempt to omit comments where duplication occurred in the groups.

The list has since been arranged as to general categories for easier reference.

CHEMISTRY

- What are the restrictions on reagents imposed by the plant operations; that is the chemists designing the process must be aware of problems that may result from the mineralogy of the ores or the process variables required
- Need a reagent for Co extraction at about pH 1.5, as in the case of Cu extraction, so as to eliminate the acid neutralization. A higher cost reagent may be acceptable as the metal being recovered is of higher value.
- Research is required on reagent degradation, the causes and mechanisms occurring.
- Speciation studies are required on all solutions throughout the process, not just the solvent extraction process, if the plant operations are to be optimized; again this requires time, particular expertise and funding. Universities can be an asset in this area.
- Understanding the process as regards the fundamental chemistry levels vs. real operation and the problems; both the organic and the aqueous phases must be considered, as there are changes in the kinetics, viscosity and physical aspects as loading occurs.
- Analyses: frequency of sampling, on-line vs. off-line, modern instruments vs. old "wet" techniques
- Is there a better way to treat fouled organics other than by clay treatment, which is expensive as well as little understood as to functionality and may not always be completely satisfactory.

CHEMICAL ENGINEERING

- # Requirement for more sophisticated design models of equipment, eg., columns

- # Modelling/simulation
  a) can it help in the plant design?
  b) possible use in the total plant system

- # There appears to be a lack of data on the thermodynamics in concentrated solutions, as most basic work has been performed and published on dilute solutions; but funding is required.

- # Application of columns to metals such as copper--will depend on the kinetics; can the rates be increased?

DESIGN

- # Use real solutions for design; difficult to relate from one plant to another and so the physical characteristics as well as the chemical will differ. Also, once the plant is in operation, the performance may differ compared to the bench-pilot plant data due to perhaps the accumulation of surfactants. Thus the typical plant solutions should be used.

- # SX process operation is dynamic and changing continually in physically and chemical compositions, (including surfactants) during the plant life, compared to the original process design based on a static sampling.

- # Dependence of design on the aqueous media, eg. SO_4^{2-}, Cl^-, NH_4^+ as regards reagent degradation, contactor choice, physical dynamics, final product, economics etc.

- # Fundamentals of chloride leaching systems, or other novel systems could be considered.

- # Piloting usually is performed using mixer settlers, from which the data is taken for scale-up; however the mixer settler used will be quite different in design from the eventual plant. Very little interest has been on other possible contactors, such as columns, where the piloting would be performed on a similar engineered design as the final plant.

- # Piloting should be considered in the full context of the total (metallurgical) flowsheet, so that leaching is really part of the pilot plant.

- # Interfacing the upstream/downstream compatibility together with the economics and the environmental impacts is important.

OPERATION

- What information is required by the operator in order to operate the process in the most efficient manner.

- How much data has to be provided to the design engineers, how long should a pilot plant be run, and what data are really required.

- Guidelines are required regarding entrainment (mechanical & chemical), the causes and the effect of tip speed, shear, etc. on organic entrainment as well as aqueous entrainment.

- There is a need for sensors that are robust to the process.

- Start-up problems, as seen by the designers, operators, engineers; good partnerships are necessary between the engineering company who built the plant and the operator regarding the plant performance. Plants should be designed to be easy to start-up and have easy access to all areas and be "operator friendly".

- Linking of 2 SX circuits can pose different, and often, difficult problems (Mo/Cu, Ni/Co, Co/Cu, Cu/U); more work is required in prediction and design in the pilot through to the plant operations rather than discover the problem after the plant has been in operation.

- Do we have all the necessary plant data and process monitoring requirements throughout the plant and operation?

- Reagents conservation should be considered by recycle of waste streams where possible, but recognizing the role of minor impurities and the possible effects on the mechanical aspects (rubber liners), or of the metallurgy.

- Analysesof components/impurities/entrainment by on-line or off-line must be considered; also includes solubility of organic components as well as the degradation of organic components of the solvent mixture.

- Sampling, and the concern with integrity of the samples, which can vary over time, etc; realistic samples are required.

- Precipitation/ crystallization from either one or both phases can occur during the plant operation, such as the basic nickel sulphates in the ammoniacal systems.

- Diluents/modifiers effects not only on the mass transfer and physical aspects, but also the effects of possible additives to the diluent due to possible changes in the legislation (limit addition of aromatics); no reliable analytical methods exist.

- # Revision of design parameters in light of "recent" knowledge regarding better design of the process; which are the real important parameters?

- # Organic removal from aqueous phases: consider equipment, reagent "cleaning", and regeneration; what is required for the removal and what is the effect on the economics? Better methods have to be devised.

GENERAL

- # SX-EW is considered as "known technology" by many operators, but not by all the industry.

- # Communication between different groups (both ways) required.

- # SX is considered by the banking society as "exotic"! Together with bad publicity, funding for major projects becomes difficult, as the financial houses do not understand the SX process.

- # SX vs. environment, where SX is perceived as an environmental risk because of the general public understanding, and the "green process technology" which would eliminate the use of solvents.

- # Safety/health concerns include the removal of the word "solvents" from processing; education and improved public relations are required. Biodegradability and toxicity must be addressed. Do we need better methods and equipment for entrainment?

- # Deficiency of published information (eg. handbook) that could address real issues, as compared to most publications which may be little more than literature reviews. Something like a small book for the operators of pilot plants and plants.

- # Differential funding (R&D vs. pilot). The research funding is usually easier to attain.

- # Generic funding for research is practiced by such as AMIRA (Australia), MIRO (UK-Europe), Copper Research Association (USA), Separation Processes (UK), NRC (Canada), and others which should be identified.

- # Considerable information and data are available in the literature, mostly from academia, which needs to be "translated to the plants" so as to be more useful.

- # Novel applications of SX include catalysis and the production of powders with defined size and other qualities

WORKSHOP SUMMARY RECOMMENDATIONS

The smaller Workshop groups identified certain items of concern, from the original list of general concerns, pertinant to their technical/scientific area. Those items were discussed in depth where possible, and the summaries are given below.

CHEMISTRY

1) Reagents

- # The chemistry in the organic phase is often different from that in the aqueous phase, and thus use of novel organic chemistry in extraction.
- # Hydration of organic species is related to the nature of entrainment.
- # “Chemistry" research has been most related to dilute solutions, while "process" operation is in the region where micelles exist; thus the requirement to work on more concentrated solutions.
- # Work on "real solutions", and don't exclude stripping.
- # Acidity depends on the H^+ activity which varies with ionic concentration; this varies in concentrated aqueous solutions.
- # Speciation of mass transfer complexes does not always correspond to those governing distribution, (eg. pH effect).

2) Degradation

- # Degradation is system dependent.
- # Plants should be encouraged to put solvent treatment stages into the circuits.
- # Examples of different systems discussed, eg.,
  - a) generation of Cl_2 and MnO_4^- in the tankhouse
  - b) storage of protonated tertiary amine which can be converted to secondary amines, and thus extract Fe.
- # Degradation may affect the chemistry as well as the physical operation.

- # Solvent regeneration process not very imaginative; eg.,
  - a) clay treatment
  - b) anti-oxidants
  - c) sodium carbonate treatment
  - d) distillation of the organic phase

3) Diluents

- # Role of the diluent least understood; more basic work required on the reagent/diluent behaviour.

- # Aromatic content vs. performance required.

- # Problem of environmental impact plus a bad image of organic compounds exists.

4) Analysis/Monitoring (in-line; on-line; off-line)

- # In-line systems are required for continuous analyses inside the stream, although on-line are acceptable. Off-line is where the sample is sent to a lab for determination and is thus not desirable for process control due to the time to receive the analysis. Some examples of on-line are:
  - a) entrainment
  - b) solution composition characteristics
  - c) organic phase analysis

Techniques include: XRF/AA/Spectroscopy/Density/Viscosity

For process control, the exact concentration is not really required, only changes or variations in concentration.

5) Novel Systems

- # Microemulsions

- # Non-dispersed solvent extraction (membranes and barrier between organic and aqueous phases).

- # Liquid membranes (supported membranes, emulsion membranes).

- # Extraction chromatography.

Conclusions: would be useful in the future in niche applications, but do not see these systems replacing solvent extraction. They also still need considerable R&D.

Limited applications: high value products (pharmaceutical)

Specialized area: analytical chemistry

Supercritical extraction: (non-metals)

6) Environmental Aspects/Image

- The word "solvent" has a bad image and there is a need to sell the positive aspect.
- Development of a "solvent-less" technology, although this is probably not possible.
- Alternatives to solvent extraction should be costed on a full life cycle analyses (often the alternatives are worse as regards environmental aspects).
- Competition from distillation in organic chemistry.

CHEMICAL ENGINEERING

1) Mass Transfer/Kinetics for Multi-Component Systems

- # Models of multi-component systems are required, together with mass transfer and kinetics on real solutions.
- # Acquisition of pilot plant data
- # Prediction of rates for design for mixer settler and columns
- # More realistic diffusivity data and models for diffusivity coefficients are required, based on the activity coefficients for multi-component systems.
- # Methods of interpreting effects of contaminants at interfaces
- # Phase continuity effects must be established in the system.
- # Laboratory scale test equipment for generating engineering data.
- # Effect of degradation products on the chemical engineering and mass transfer

2) Equilibrium/Thermodynamics

- # Data collection and better prediction methods are required for real systems, based on thermodynamics.
- # Research is encouraged to define the computer codes to enable optimum use by the design engineers.
- # Species identification necessary.
- # Fluid structures, such as micelles and interfaces
- # Systems containing water

3) Design Models

Prediction tools are required for first-level design of columns and mixers based on drops.

More data are required on coalescence and breakage coefficient, which would be used in conjunction with pilot plant data.

Rate of sedimentation of drops in swarms at high phase fraction for a range of physical properties and energy input to be determined.

Computational fluid dynamic models (CFD) at present are not promising for column design in hydrometallurgy. Problems to answer include:

a) couple effect of energy dissipation;
b) inclusion of drop population equation to describe drop dynamics**;** and,
c) incorporate interphase mass transfer with reactions in the drops and in the continuous phase.

Encourage researchers to design computer codes that are user-friendly for the design engineer.

How to verify computer-aided calculation procedures.

Laboratory scale data acquisition

Financial support is required for research.

Education/collaboration with industry (need geometry of plant, physical properties, equilibrium information, feed and raffinate concentrations) are required.

4) Process Dynamics/Control Start-Up

Control of processes with drift in chemical properties of solvent and solution over time

Classical control principals are useful on a day-to-day basis.

Models are available on drop population balances for multi-variable control.

Tailored solutions are required for small batch processing of exotic separations.

Option to use small continuous extraction columns

5) Scale-Up

- # Collaboration between academia and industry desirable.
- # Validation of computer models require specific information from column designers and the manufacturer to provide large scale-up data.
- # Provision of a data bank of useful, non-proprietary data for scale-up required.
- # All types of solvent extraction contactors should be considered in the design.
- # Drop coalescence and break-up phenomena; flow patterns can change with scale-up.

6) Monitoring

- # Sample integrity and obtaining relevant data
- # Principal component analyses are useful for complex systems.
- # Use of models are helpful, and the understanding of basic principles are necessary.
- # Robust monitoring techniques are required and should be implemented in the process, eg., ion selective electrodes, ultrasonic monitors, densitometers, on-line spectroscopy.
- # On-line analytical equipment desirable.

7) Entrainment/Crud/Solvent Maintenance

- # More work is required to determine the minimum drop size generated by various impellers over a range of physical properties and energy input.
- # The presence of micelles may be the cause of many problems being encountered in producing stable emulsions and cruds.
- # Flow patterns in vessels which could lead to entrainment need to be modelled.
- # A broad scope study is required to define the physical characteristics of emulsions (L-S) leading to crud formation.
- # Studies are required on crud formation and de-stabilization.
- # Use of CFD to understand entrainment problems
- # Large scale equipment studies where possible

8) Novel Systems

- # Supported membranes

- # Equipment for application to systems operating at extreme phase ratios (eg. 10/1). Problems of generating small drops in mixer settlers, so other contactors have to be considered. (eg. film contactors and perhaps hollow fibre membranes).

- # Supercritical fluid extraction is a possibility, but economics may be a deterrent. More work is required.

- # Active adsorbent as particles or in membrane form

- # Speculative areas:
  - a) micelles for extractive separation
  - b) 2-phase aqueous separation (biochemical applications)

- # Application to 3-phase systems (L-L-S)

9) General Comments

- # Education and challenging projects.

- # Communication with industry and engineer/researcher/politician interface.

- # Continuing education courses, major conferences and Workshops.

- # Liquid-liquid extraction instead of solvent extraction if "solvent" is a perceived problem.

- # High turnover of personnel throughout the industry, and greater pressure on the universities; really a post-graduate activity at the universities rather than an undergraduate activity.

- # Incorporate knowledge into software and expert systems to transfer to industry.

- # There is a need for a text for the industry that has the practical applications, not a large handbook that may be too scientific.

- # A general lack of funds often makes research difficult.

PROCESS DESIGN, PLANT DESIGN, AND OPERATIONS

1) Models

- More sophisticated models are required in the areas of:
  - a) equipment (design and operation methodology for system performance optimization)
  - b) process (operating variables and scale-up)
  - c) fundamental

- Shortfalls of existing models
  - a) practicality of using academic models
  - b) physical aspects (eg. dispersion/coalescence) to be considered in the models
  - c) engineering application (use)
  - d) better definition of requirements of the model by the engineers
  - e) lack of settler models
  - f) limits of the models
  - g) lack of fundamental data for the model optimization
  - h) validation of the model

2) Contacting Equipment And Operation

- Equipment selection
  - a) consider the chemical aspects (mass transfer, kinetics) and the physical operation (dispersion/ coalescence, emulsions), eg. pump-mix vs. pumping and mixing; in-line mixers, or other contactors ----all to generate less shear by better control of the mixing.

  - b) plants should be designed for the flows that are anticipated, not over-design to allow for increased flows in the future. Extraction time in the mixer should be based on kinetics, and mixing for longer times than required results in increased small droplets which cause poor phase disengagement.

2) Piloting to Scale-Up

- Feed characteristics(composition, flow)

- Process chemistry, equilibrium data

- Use kinetics in the design, rather than over-design in the mixer, so as to achieve better discrimination over impurities as well as reduce the mixing time to the minimum rather than a high shear mix over extended times, resulting in micelles and cruds.

- Introduction of purification (scrub) stages where possible.

Choice of the contactor based on the chemical equilibria and kinetics, and dispersion/coalescence characteristics. Various types of contactors should be considered, not just mixer settlers as in the past, but also to include columns.

Phase separation and settling tests, if possible in a special unit such as a deep settler.

Continuous testing and piloting and the assessment of physical characteristics are required for each specific plant; other data from other similar plants or other metal processes are not really relevant.

Objectives of testing on a continuous circuit have to be clearly defined so as to obtain the most information from the tests.

The size of a pilot plant will depend on the company objectives of proving the process, obtaining products, and having a demonstration plant. They have varied in size from a 5000-mls per hour to a 100 $m^3/m^2/h$. Of course there will be a considerable difference in the hydrodynamics over this range. If time and resources permit, 2-3 different size pilot plantswould be desirable. Pilot plants are no longer built to the 1/10 plant size of years ago.

Duration of pilot testing will depend on the objectives (chemistry, physical, bankers, etc) as well as problems that may not have been anticipated in small-scale work, such as cruds/precipitates/solvent degradation, effect of recycling and impurity build-up, etc. That is, integrate the "upstream and downstream" processes.

Sound scale-up methods

Materials of construction in the small-scale pilot plant may be quite different as compared to the eventual plant. Therefore scale-up could be difficult. This important aspect should be considered in the pilot testing and in the acquisition of scale-up data.

Consider economics relevant to the capital and the operating costs (often the high daily operating costs will kill the operation, whereas the capital costs can be amortized).

Process guarantees--many plants have been designed based on what has been done before, not on what are probably improved designs which have not been in great use.

3) Start-Up Problems

- Need more sharing of experience; very little has been published on the problems that have been experienced. The Mn contamination in Cu plants in Australia is a recent example.
- Poor piping design; insufficient holding volumes when some mixer settler plants are shut down and settlers flood.
- Materials of construction and compatibility are often concern (gaskets, pipe).
- Operator access often poor as regards to valves, mixers, settlers, etc.
- Gravity line design
- Air entrainment often high in the mixer settlers, both in the flow to the settler from the mixer, and also organic entrainment in aqueous from the weir. Maintain the launder full.
- Quality of construction
- Chemical differences between test solutions and actual solutions, as well as the processing of the actual ore body and not a small sample.
- Operator ability and training are important, and will vary with the relative remote locations and personnel availability.
- Start-up engineers must be able to communicate with the operator
- Re-evaluation of start-up by the engineering company desirable.
- Continuity of design and start-up
- Availability of spare parts
- Personnel turn-over 3%-20%, or higher
- What to do with off-specification materials

4) Operational Information Required to Operate the Process

What are the actual operating parameters limits

Entrainment measurements (on-line) for the specific stream

Reliable sampling and analyses (on-line, off-line); some on-line methods have proven excellent (eg AMDEL) for uranium

Solution composition and its characteristics regarding possible emulsion or precipitation tendencies

EMF analyses

More information at extremities such as the feed concentrations into the circuit and solutions leaving the SX circuit.

Phase continuities and the effect on the chemistry of the process as well as the physical aspects

O/A ratios dependent on the (changing) feed composition, and the recycle phase

More organic monitoring and analyses for performance (loss of reagent, poisoning due to organic acids or other foulants, or degradation)

Crud characterization, and analyses

5) Entrainment Reduction (mechanical/chemical)

What is good operating practice (Cu circuit 10-50 ppm; U circuit 20-200 ppm); obtain a representative sample (obtain several litres and measure), and analysis.

Aqueous entrainment in organic at typical Cu operations (400 ppm) also a serious problem and can be affected by phase continuity.

Analyzing the problem in the plant. Entrainment is often a costly problem. Inventory is a guide to the amount lost if analysis prove difficult. Sample bottle often a problem because of adsorption. Gas chromatography is used on the organic entrainment in aqueous determination.

Some releases monitored and tested by toxicity tests prior to release.

- # Decrease in entrainment accomplished by such as an after- settler, increase temperature to 50-55 °C, and aerate which releases the solvent.

- # Equipment sizing, selection and operation (columns, mixer settlers/mixer type) relative to the process; different entrainment will result from different mixer settlers, and columns have shown to result in lower entrainment.

- # Coalescence media in settlers and on exit streams have proven successful; however the coalescing media can also be crud traps and cause operating problems. Flotation columns have been used with varying success (produce down to 20 ppm); may be useful coupled with dual media filters, but coalescers would be the preferred.

- # Specific flowrates across the settler

- # Organic operating depth in the settler

- # Question of having a loaded organic tank and barren organic tank (entrainment removal)

- # Solution velocities

- # Extractant / diluent choices can affect the phase separation as well as the amount of entrainment.

- # Operating philosophy on losses that may be tolerated varies with country and process.

- # Influence of air on entrainment, due to the design and the operation of the particular contactor, especially in mixer `settlers.

- # Phase continuity control a problem in most mixer settlers, but not a problem in most columns.

<u>6) Organic Removal from Aqueous</u>

- # Need more information on how to achieve low levels of entrainment
- # Cu-foil industry has experience
- # In-settler coalescence media
- # Codelco/Church coalescers
- # Knit mesh for coalescing
- # Extraction with another solvent which is then stripped
- # Low cost adsorbents
- # Distillation
- # Scavenger circuits
- # After-settlers; air flotation

<u>7) Reagent Degradation</u>

- # Type of degradation is system dependent
- # How is extractant destroyed:

  a) UV- cover the settlers or use columns if kinetics favourable)
  b) high oxidant levels (eg. metal oxidants, reagent oxidants such as nitrates, chlorine)
  c) temperature
  d) conc. acid addition to organic phase produces degradation products and surfactant which cause phase separation and entrainment problems
  e) inappropriate storage and/or long storage, eg. tertiary amines stored in the protonated form caused degradation to secondary amines
  f) bacteria

- # Degradation of diluents and modifiers (bacteria can degrade both components, and aliphatic components at probably a faster rate than aromatics). Isodecanol degrades to isodecanoic acid to result in surfactant problems). Oxidation reactions can affect both components.
- # Aromatic content vs. performance (environment vs. degradation). A certain amount of aromatic content may be necessary in order to prevent bacteria problems as well as reduce any phase separation problems.

- # Equipment design for inert gas atmosphere operation may be required; columns and in-line mixers could be possible solutions to the problem depending upon the kinetics of the particular system.

- # Poisoning of organic phase (not actually degradation but loss of active sites, such as the presence of organic acids (humates, degradation of alcohols, recycle of aqueous effluents from tailings ponds that also contain the sewage disposal).

- # Degradation products and effect on recycle

- # Reversible Degradation (Regeneration)
    a) loading capacity
    b) scrubbing/stripping efficiency
    c) phase separation
    d) selectivity
    e) reaction kinetics
    f) crud formation
    g) contamination in sequential circuits
    h) analytical error

- # Methods of Regeneration/Prevention (removal of surfactant)
    a) sodium carbonate regeneration (uranium)
    b) distillation of organic phase
    c) removal of degradation products (clay treatment used in Cu circuits)
    d) change of aqueous ionic strength
    e) addition of anti-oxidants to decrease degradation

NVIRONMENTAL CONCERNS

- # Soil, water, air

- # Metals, Organics
  - a) health effects of the solutions, and identifying the toxicity of the potential new hazardous chemicals; acute toxicity data available on most commercial reagents
  - b) fire hazard (and how to separate the mixer settlers of equipment from the actual fire; engineering required. Some plants with automatic discharge to an external organic sump)

- # Safe containment of wastes
  - a) within the plant
  - b) safe containment of organics and crud in the waste dump

- # Waste disposal
  - a) cruds
  - b) incineration
  - c) landfill or leach pad

- # Risk assessment analysis

- # Environmental impact data

- # Decommissioning protocols

- # Safety design in plant equipment
  - a) remote sampling/operation
  - b) ventilation - negative pressure
  - c) minimize venting as much as possible, eg. cover mixer settlers, or use of columns where applicable. Also keep sampling and viewing hatches closed
  - d) selection of the reagent-equipment-operation procedures to reduce the environmental impact

- # Waste Reduction
  - a) particularly to reduce the amount of entrainment and the amount of crud. The optimization of equipment choice and its operation and control are required.

- # Oxidation of Wastes (Dissolved Organics, Entrained Organics)
  - a) organic wastes/aqueous wastes
  - b) achieved by:
    - $O_3/O_2/H_2O_2$/UV/TiO_2
    - supercritical water as an oxidizing agent
    - use of Reverse Osmosis to concentrate organics prior to oxidation process

- # SX plants in communities vs. "green fields" large metallurgical operations where the amounts of solvent, as well as the facilities for treatment/ disposal differ

- # Hazard planning
  - a) containment for diluents storage
  - b) iso-containers for extractants
  - c) consider portable units that could be applied to specific treatment for organic recovery for re-use

- # Biodegradability vs. performance

- # Life Cycle Analysis (cradle to grave)
  - a) "green technology"
  - b) zero discharge

CLOSING REMARKS - Gordon M. Ritcey

I believe that this has been a successful Workshop in the time that we were here in Banff. This was the first such Workshop, and I hope that this format for discussions will continue. Certainly the response to this one has been very positive. The interest generated in this Workshop indicates that similar meetings should be held periodically to address the problems and concerns of the Solvent Extraction process.

Before closing the Workshop, I want to thank and acknowledge:

a) the International Committee for Solvent Extraction Technology for sponsoring the event and providing the "seed money";

b) the helpful discussions I had with many of you related to the program and planning;

c) the assistance of Geoff Stevens and the University of Melbourne for receiving communications for me while I was travelling in Australia earlier in 1997, and also for mailing out the last information;

d) the generosity of Cytec and Bill Rickelton for providing such splendid brief cases;

e) the help of several who kindly assisted at the registration desk: Geoff Stevens, Lucky Lakshmanan, Manuel Valiente, Dean Thibault; Peter Carlin, Noel Hayward, Ron Molnar.

f) the help of Ron Molnar and my wife Gladys in unpacking the briefcases and placing the material inside;

g) the several co-chairmen of the Sessions: Mike Cox, Philip Horwitz, Mike Virnig, Mike Slater, Larry Tavlarides, Geoff Stevens, Nevil Rice, Ron Molnar, Lucky Lakshmanan, Henry Schnell, Mark Vancas, Jim Lommen;

h) the Keynote Speakers for their presentations and for providing background necessary to the subsequent Workshop discussions;

i) to J.F. Tremblay, Conference Director of the Banff Centre, who was my contact over several months of planning and his help with the arrangements for the Workshop; and

j) finally to you, the participants, who took time out from your busy schedules to participate in this Solvent Extraction Workshop'97.

APPENDIX

QUESTIONNAIRE/COMMENTS ON SX WORKSHOP'97

The following is the summary of replies to the questionnaire to the participants of the Workshop.

RATING OF WORKSHOP RELATIVE TO MY REQUIREMENTS
% Distribution

EXTREMELY USEFUL AND INFORMATIVE	60
INFORMATIVE	32
INTERESTING	8
WASTE OF TIME	

PARTICIPATION IN DISCUSSIONS

I PRESENTED A KEYNOTE	24
I PARTICIPATED FREQUENTLY IN THE DISCUSSIONS	48
I PARTICIPATED OCCASIONALLY IN THE DISCUSSIONS	42
I PARTICIPATED VERY LITTLE IN THE DISCUSSIONS	10

WOULD YOU ATTEND A SUBSEQUENT WORKSHOP?

YES	97
NO	3

WORKSHOP PROGRAM AND FORMAT

I LIKE THE IDEA OF EVENING SESSIONS WITH THE AFTERNOON FREE	79
I PREFER TO HAVE THE EVENINGS FREE	21

I THINK THE DURATION OF THE WORKSHOP WAS:

SUITABLE	90
TOO LONG	3
TOO SHORT	7

IDEAL NUMBER FOR SUCH A WORKSHOP

50	75	75-85	100
11	44	28	17

CONTENT OF WORKSHOP

ARE KEYNOTE PAPERS REQUIRED?	YES	92
	NO	8

VENUE AND FACILITIES

	EXCELLENT	GOOD	POOR
WORKSHOPS	13	87	
MEALS	14	38	48
RECREATION	78	22	
LOCATION	94	6	
ACCOMMODATION	69	31	

MEALS

FOR SUCH A GROUP, THE MEALS BUFFET PACKAGE WAS IDEAL	86
I WOULD PREFER TO PLAN AND ORDER MY MEALS	14

DO YOU THINK A SPECIAL DINNER IS REQUIRED?	YES	19
	NO	81

WAS THE RECEPTION ADEQUATE?

IN LENGTH	YES	93
	NO	7
IN CONTENT	YES	96
	NO	4

SOCIAL ACTIVITIES

IN A FUTURE WORKSHOP I WOULD:

BE INTERESTED IN PLANNED ACTIVITIES (EG. TOURS, HIKING, FISHING, RAFTING, ETC)	34
PREFER TO ORGANIZE MY OWN ACTIVITIES	66

OBSERVATIONS/COMMENTS/SUGGESTIONS

The following is a list of all the comments by the participants:

<u>GENERAL</u>

best and most useful SX meeting attended

more useful than ISEC meetings in some ways, eg. critical discussions were valuable; potential to be extremely useful

many useful ideas were developed

the Workshop should be a regular event, again of limited invitation

interaction of disciplines and industries was good

essential to maintain the diversity: geography, R&D, operators, contractors, etc.

a gap still exists between scientists and engineers

<u>FORMAT</u>

with a location like Banff, the afternoons free was best; if in a city, perhaps best to have evenings free

evening sessions could begin earlier and terminate earlier

one arranged dinner would be useful early in the Workshop

option to have planned and ordered meals

meal package of breakfast and dinner; breakfast and lunch

one free evening out for meal

one guided tour on first day to keep participants together

need 2 breaks during evening sessions

require more time for discussion between delegates

unfortunate that academics and operators were not together more

- # keynotes could be placed within the sessions
- # discussion within the workshop groups was the most effective
- # one group activity would have been useful

KEYNOTES

- # keynote papers help to form the basis for discussion
- # fewer keynotes; perhaps only a couple before each session
- # all keynotes should be reviewed before presented
- # only 1 keynote for each of the sections of: chemistry, chemical engineering, process development, plant design
- # pure science should be related to applications; less theoretical presentations
- # keep presentations to 15 minutes
- # keynotes should be oriented towards areas of knowledge/experience which are lacking
- # solid supported ligands should not be included in such a workshop

SUBJECT AREAS

- # scaling and piloting of columns
- # basic design (from operating point of view)
- # problems in operation
- # more practical operation data would be helpful
- # more discussion on operation problems
- # include areas of biotechnology, organics, pharmaceutical,chemical process industry, electronics & high performance materials
- # more detailed discussion of actual projects/problems/solutions
- # automation

- process robustness
- areas that were covered were excellent for a first workshop; future workshops should delve more deeply into specific subjects
- useful to have senior management perspectives of large operating companies towards the future
- consultants view of various industries
- have the "brainstorming" early and omit the keynotes
- concerted effort by industry needed to apply the knowledge of R&D
- a workshop on Plant Development and Operation is suggested
- funding opportunities not covered adequately

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